

# Investigation of Fly ash Polymer Composite



Thesis submitted in partial fulfillment of the requirements for the degree

of

*Master of Technology*

in

**Metallurgical and Materials Engineering**

**Submitted By**

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**Department of Metallurgical and Materials Engineering**

**National Institute of Technology**

**Rourkela-769008**

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Under Supervision of

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2014



## National Institute of Technology, Rourkela

### Certificate

This is to certify that the thesis entitled, “**Investigation of Fly ash Polymer composite**” submitted by **Mr. Satyajeet Kumar** in partial fulfillment of the requirements for the award of the degree of **Master of Technology in Metallurgical and Materials Engineering** at the **National Institute of Technology, Rourkela** is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any degree or diploma.

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Finally, I feel great reverence for all my family members and the Almighty, for their blessings and for being a constant source of encouragement.

**Place: Rourkela**

**Satyajeet Kumar**

*Dedicated  
To  
My Beloved  
Family*

*Whose blessings, love and sacrifice brought me here up to .....*

**ABSTRACT**

Industrial waste like fly-ash which is creating environmental problems, is mainly used as a building material due to its low cost and easy availability. But the main disadvantage of these bricks is its low strength. So, a lot of research is going on to increase the strength of these bricks. The present research work is carried out to develop a new systematic procedure to produce fly ash composite bricks which will have higher compressive strength. Here the fly-ash is mixed with Cold setting resin at different proportions and water treated at different temperatures to find out a solution to the brick industry. The compressive strength, Hardness, water absorption, Density and thermal conductivity of the fly ash-resin powder bricks obtained under optimum test conditions are 11.24 MPa, 47.37HV, 19.09% 1.68 g/cm<sup>3</sup>, and 0.055 W/mK respectively. The sliding wear behavior is also investigated. The structure-property correlation of these composites are studied using X-ray diffraction, FTIR analysis and scanning electron microscopy.

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## ABBREVIATIONS

<b>FA</b>	Fly ash
<b>BA</b>	Bottom ash
<b>EPRI</b>	Electric Power Research Institute
<b>ACAA</b>	American Coal Ash Association
<b>MOEF</b>	Ministry of Environment and Forest
<b>LOI</b>	Loss on Ignition
<b>IRC</b>	Indian road Congress
<b>FAM</b>	Fly ash Mission
<b>CSH</b>	Calcium Silicate Hydrate
<b>CASH</b>	Calcium Aluminate Silicate Hydrate
<b>ASTM</b>	American Society for Testing and Materials
<b>FESEM</b>	Field Emission Scanning Electron Microscopy
<b>SEM</b>	Scanning Electron Microscopy
<b>XRD</b>	X-Ray diffraction
<b>FTIR</b>	Fourier Transform Infrared Radiation
<b>JCPDS</b>	Joint Committee on Powder diffraction Standards
<b>NTPC</b>	National Thermal Power Corporation
<b>CPP</b>	Captive Power Plant

A blue geometric graphic consisting of several overlapping triangles and quadrilaterals, creating a dynamic, abstract shape that tapers to the right.

# CHAPTER 1

# Introduction

## 1.1 INTRODUCTION

The entire development of a country depends on the production value of power and consequently its consumption as energy. Our country, India needs huge power resources to meet the expectation of its occupant as well as its aim to be a developed nation by 2020. Fossil fuel plays an important part in meeting the demand for power generation. Coal is considered to be one of the world's richest and widely distributed fossil fuel. Around the world, India dominates the third position in the largest production of coal and has the fourth largest coal reserves approx. (197 Billion Tons). It has been estimated that 75% of India's total installed power is thermal of which the share of coal is about 90%. Nearly about 600 Million tons of coal is produced worldwide every year, with Fly ash generation is about 500 MT at (60-78 %) of whole ash produced [1, 2]. In India, the current generation of FA is nearly about 180 MT/year and is probable to increase about 320 MT/year by 2017 and 1000MT/year by 2032 [3]. No doubt Indian coal has high ash content and low heat value. In order to meet the increasing challenging demands, many coal based thermal power plants have been constructed. As a result of which huge amount of combusted residue in the form of Fly ash (80 %), and Bottom ash (20%) has been produced. The finely dispersed particle from the burnt coal is discharged out through the flue gases which are detached mechanically through electrostatic precipitators and separators which are then collected together in the field of hoppers. The rate of production of FA is high and it goes on increasing year after year. The annual production of FA in China, India and US is approximated about 275 million metric tons. But less than half of this is consumed in various areas. The greatest challenge before the processing and manufacturing industries is the disposal of the residual waste products. The harmful impact on the surroundings suggests the necessity for appropriate dumping of fly ash and justifies full utilization of FA when feasible. Waste products that are generally toxic, ignitable, corrosive or reactive have detrimental environment consequences. This major issue requires an effective, economic and eco- friendly method to tackle with the disposal of the residual industrial waste products. The problem with safe disposal of ash without affecting the environment, disturbing ecological balance and the large storage area required are major issues and challenges for safe and sustainable development of the country. Hence needful efforts are being made continuously by making stringent regulations by the government to fully utilize the ash. Currently only 50% of the fly ash is being profitably utilized in India [4]. The most common and feasible ways to utilize these industrial wastes products is to go for construction of roads, highways and embankments. The Problem with environmental

pollution can be greatly reduced if these wastes products be effectively utilized in construction of roads, highways and embankments. But sufficient amount of soil of desired quality is not available easily. So these industrial wastes not only used as an alternate for natural soils in the construction rather it also solve the problems of disposal and environment pollution. This will provide a number of significant benefits to the constructing industry as well as to the country as a whole by conservation of natural resources, by reduction of volume of waste to landfills, by lowering the cost of construction materials, and by lowering waste disposal costs. With the help of some suitable stabilizer like lime, thermosetting resins or cement, the properties of fly ash can be increased and it can be further used as a construction material. FA shows self -hardening behavior that is why it is used in construction broadly.

## 1.2 Fly Ash: An Overview

FA is an Industrial waste which is accepted as an environmental pollutant, generated during the combustion of coal for energy production. When the coal is fired inside the grate of a boiler, Carbon and volatiles materials completely burnt off. But still, some inorganic impurities of earth elements (sand, Feldspars etc.) are bonded together and are discharged out through flue gases. When these fused materials are allowed to solidify, it results in the formation of fine and spherical particles called Fly ash. These FA particles are tiny spheres enclosed in a big sphere called plerospheres. Hollow spheres are also called cenospheres. The morphology of FA particles is sphere due to the bonding which takes place during suspension of released flue gases from chimney or boiler. These fine particles mainly consist of oxides of silicon, aluminum and iron. Some elements like P, Mg, K, Ca, with small traces of Cu, Zn, Mn, Fe, B, and Mo are also found. The properties of FA vary from different sources, from the same source but with time and with the techniques used for handling, storage and variation in load generation [5].

Fly Ash can also be known by its different name such as Coal ash, Pulverized Flue ash, and Pozzolona. FA is characterized by its light weight, Silicate glassy appearance, spherical in shape, grey colored, polymeric, alkaline and refractory in nature. In addition, FA has a pozzolanic property [6]. The FA forms a hard and cementitious compound like calcium silicate hydrate and calcium aluminate hydrate in the presence of moisture. The hydration reactions of fly ash and Portland cement are almost similar resulting in similar properties as compared to each other. It is advantageous to use FA as a replacement of cement in the concrete, providing some distinct

features. Some of the important characteristics shown by fly ash concrete are better textural consistency and detail of sharper. Fly ash shows similarity with volcanic ashes which was used to produce hydraulic cements almost 2,300 years before. The term "pozzolans" was named after a small Italian town of Pozzuoli where these cements were made. A pozzolan is a silica and alumina rich material which forms a hard and cementing compound in the presence of moisture. The features of Pozzolans and lime binding capacity of fly ash makes it possible for the production of high strength bricks, cement and aggregates [2]. One of the best pozzolans in the world is fly ash and that is why it is best known with this. Now a days fly ash can directly be taken from coal fired power generation plants, so Fly ash coming from volcanoes are of no use. Before combustion, these power plants grind coal to powder fineness. After burning the coal huge amount of fine residue can be collected from the exhaust of power plants and these can be used further. Both Fly ash and Portland cement appears to be structurally similar but can be distinguished under optical microscope. Fly ash particles are almost spherical, and can be allowed to move and blend freely in any admixtures. Fly ash possess excellent physicochemical and mechanical properties which includes low dense structure with high strength, negligible porosity and shrinkage, excellent thermal stability and durability, high surface hardness, and better fire and chemical resistance. Owing to these characteristics feature of FA, it can be used in different civil, mining and metallurgical applications like architectural sector, transportation and aerospace industry, as road sub base material, wear resistant ceramics and tiles, Geopolymers and many others. There is an extensive variation in the physical and chemical configuration of Indian FA. These variations are mostly due to the combustion chamber or incinerator efficiency. All the thermal related plants of India are governed and accomplished by a single unit i.e. NTPCs. Unavailability of decent quality of coal, below standard maintenance and non-renewal of different parts of combustors even after the completion of its ideal life are some of the features accountable for low incinerator efficiency [7].

### **1.3 Fly Ash Bricks**

Bricks has been used as a major construction and building material. Since long Aluminous –silicate and silica bricks are chosen as refractory materials in many industrial applications, due to their high wear resistance, long lasting, sturdy and load bearing capacity at high temperatures [8]. Due to the limitation of clay resources, china has partially restricted the use of conventional fired bricks

produced from clay [9]. Therefore the ultimate aim is to find raw materials for brick production alternative to clay. These days energy savings has become a very important environmental and economic issue. The consumption of energy from buildings comprises about one third of the total consumption, with nearly half of its energy lost through the walls [10]. One of the effective approaches to reduce energy consumption is to decrease the thermal conductivity of wall material, such as brick. Organic residues such as saw dust, polystyrene, paper sludge, coal, coke and inorganic products are commonly used to decrease the thermal conductivity of the brick. These residues used as a pore forming additives to obtain highly porous bricks. Numerous studies have been conducted on fired brick made of Fly ash [11, 12]. FA bricks show better mechanical and physicochemical properties which includes low dense structure with high strength, negligible porosity and shrinkage, excellent thermal stability and durability, high surface hardness, fire and chemical resistance than conventional earthen bricks [13]. These bricks are an environment friendly cost saving building product. Fly ash bricks are durable, have low water absorption (8-18%), less consumption of mortar, economically stable and no emission of greenhouse gases. These bricks remain static and are not affected by environmental conditions, thus ensuring longer life of the structure. The bricks made of FA are three times resilient and stronger than conventional bricks with constant strength. Due to the presence of free lime the strength of compacts is accelerated at high rate. Hence these bricks are perfectly fit for internal and external loadbearing and non-load bearing walls. To determine the compressive strength and microstructure of the cracked samples, compacts of Fly ash and cold setting resin along with hardener with various percentages are prepared and treated in water at 110<sup>0</sup> C -180°C for 24 hours.

Salient features of FA bricks:-

- (1) Practically no damage can be seen during transport and use, due to their high strength.
- (2) Owing to uniform size of bricks mortar required for joints and plaster reduces by almost 50%.
- (3) The seepage of water through bricks substantially reduces due to its low water penetration.
- (4) Like conventional clay made bricks, FA bricks are not soaked in water for 24 hours before use. Only sprinkling of water is sufficient.
- (5) No need of Plastering.



### **1.4 Cold setting resin: - An Overview**

Cold mounting compound resin is used as a binder material to provide inter particle bond between the FA particles and to increase their strengthening effect. They are good resistance to atmospheric and chemical degradation. Resin powder cannot shows its effect alone until it is mixed with hardener (or accelerator) to provide the mounting compound, and then the polymerization process takes place to form the desired block. This process sometimes generates heat but this generation can be minimized by the use of cool air or cooling water. These compounds can be ideally chosen for those materials which show sensitivity towards heat or pressure. This cold setting resin offers better properties for Fly ash compacts. Improved mechanical strength and hardness, resistance to atmospheric and chemical degradation, reduced thermal conductivity, eliminates porosities and cavities, fast curing of compacts are some of the common properties. The setting compound and the hardener were supplied by Geosyn pvt. Ltd. Kolkata.

### **1.5 Objective of the Present work**

The aim of the present work is to fabricate Fly ash polymer composite at different proportions of polymer and to study physico mechanical, thermal conductivity and wear behavior. In present project an attempt was made to increase the density and hardness of the water cured cylindrical samples. SEM, XRD and FTIR analysis were also done to investigate the microstructural changes.



## **CHAPTER 2**

# **Literature Review**

## **2. Literature review**

### **2.1 Introduction**

The total installed capacity of electricity generation in India is about 100,000 MW. Out of which 73% of power generated is from thermal Plants. India has coal reserves of about 180 billion metric tons. Hence it is worth to say that 88 % of the thermal power plants of India utilize this abundantly found natural resource. In our country there are several power generation units out of which around 85 units are based on coal. Large amount of ashes present (35–50%) and low calorific value (2,800–4,200 kcal/kg) is the physical significance of Indian coal. To produce the required energy high coal fired rate is mandatory which thus generates greater ash deposit. In India the current generation of coal ash is nearly about 180 million metric tons; which is supposed to be two times in the upcoming decade. Generally wet method is employed in India for the ash disposal. For the production of 1 MW power it requires approximately one acre land and a better initial investment. In our country the occupancy of ash pond is almost 26,300 hectare. Till 1994 only 3% of total ash was utilized. Since then, it was realized that the environment must be conserved in India and for this our government proposed a Fly Ash Mission (FAM) in 1994. The major purpose for this mission was the safe disposal and consumption of fly ash. In India, 21 different locations are chosen to demonstrate 55 technologies by FAM in 10 major areas. The increment of the use of fly ash was recorded from 3 to 13% since 1994 to 2002. Several steps were taken by the government of India and for which MOEF (a working body of Indian govt.) delivered a proposal for better exploitation of fly ash and its consumption. According to this proposal every thermal power plants must achieve 20% consumption of fly ash in three years and 100% in fifteen years and for newly established power plants there was some liberation in the rule and they need to achieve 30 % consumption in three years and 100 % in nine years. The major use of fly ash is in compacted fills and ridges. Glogowski et al., (1992) studied the manual of EPRI and reported that with the help of fly ash 33 ridges and 31 fills were made in North America. According to the ACAA in 1999, 33% of the fly ash produced in US was used in several areas for different applications. Primarily Fly ash finds application in concrete, cement, and grout industry with (16.1%). About 5.1% of total generation of fly ash is used in structural fills. Porbaha et al. (2000) estimated that around 41% of fly ash was utilized for the production of landfills in Japan. Utilization of fly ash has got a great contribution in the field of construction of ridges and fills. The increased use of fly ash in the field of construction and fills has gained a major acceptance in technology demonstration projects.

Vittal (2001) stated that few embankments have already been constructed using pond ash in India. According to IRC, 2001 (a working body of Indian govt.) has proposed strategies to use fly ash in road embankments [14]. Fly ash shows self - hardening behavior and can be utilized in construction over wide range. This property is due to the availability of free lime .The properties of it depends on various characteristics out of which some are characterization of coal, fineness of pulverization, furnace type and temperature of firing.

## 2.2 Classification of Fly ash

Based on the amount of lime present, Fly ashes can be classified as Class F and Class C according to ASTM C618 [15], And on the basis of lime reactivity FA are categorized as Grade I and Grade II according to I.S. 3812 [16]. The type of coal burned and the amount of ash content relates the classes of Fly ash. Class F Fly ash contains low lime and ash being greater than 70 Wt. % of  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  .While On the other hand, the ash content between 50-70 Wt. percent  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  and high lime content are grouped in Class C. Class F fly ash can be produced from anthracite of bituminous coal while sub bituminous or lignite coal produces class C type fly ash. Since anthracite coal is not used for power generation so class F type Fly ash can easily be derived from this coal. Characteristics of Class F type Fly ash shows low calcium ashes having lime percentage 6, so these are not self - hardened. But normally this class exhibit pozzolanic features. The ash content from this class covers more than 2% unburned carbon which can be ascertained by (LOI) test. The major crystalline phases present in the form of Quartz, mullite and hematite identified fly ashes, derived from bituminous coal. Because of some significant properties shown by class F type fly ash, it is now most appropriate type fly ash used for research work. Cementing compounds generated by the hydration of Portland cement can also be generate by the reaction of FA produced by bituminous coal with lime or calcium hydroxide in the presence of moisture. Past research shows that class F fly ash can replace 15-30 % of cement because of its satisfactory and acceptable aggregates. The demand of water and heat of hydration can be minimized with the help of class F fly ash .It also shows high resistance to sulphate and chloride ion ingress. Class C fly ash - also known as High calcium ashes with lime (Cao) content greater than 15% became available for use in concrete industry only in the last 20 years in the 1970s. These ashes are pozzolanic along with self-cementing in nature.

## 2.3 Fly Ash Properties

Making out desirable application of FA demands the study of fly ash characterization in terms of structural morphology, interfaces between phases and its susceptibility to chemical change (reactivity) is of great significance. FA is characterized by its physical, chemical and mineralogical properties that are greatly reliant on the essence of the parent coal, the combustion conditions, various emission control devices and the storage and treatment methods [17].

### 2.3.1 Physical Properties:-

Several Physical properties help in categorizing the Fly ashes for various engineering purposes. FA comprises of fine, powdered particles mainly spherical in shape, either solid or hollow. These particles are primarily glassy (amorphous) in nature with possibly some crystalline phases. During combustion of Coal [18] oxidation occurs, therefore negligible amount of carbon and nitrogen [19] is found in the Fly ash. The color of Fly ash vary from grey to black due to the little changeable quantity of unburned carbon present in it [2]. Carbon content material in the FA are angular in shape. There is no such big difference between the variation in particle size of a bituminous coal FA and silt. Usually it is less than 0.075mm. Sub-bituminous coal FA is little bit coarser than bituminous coal FA although both are sediment-sized. The specific gravity of fly ash lies between the range of 1.8-3.2, while its specific surface area lies in the range of 180 to 1000 m<sup>2</sup>/kg [20– 23]. The average diameter of these Fly ash particles are less than 10 micro meter and size between 0.01 - 1000micrometer [16]. The Spherical morphology, low bulk density and low specific gravity bring great limitation in stacking height of fly ash and limits the height of ash dykes generated for increasing the storage capacity of ash ponds.

### 2.3.2 Chemical Properties

Fly ash properties are greatly influenced on various parameters like nature of coal, methods for handling, storage and treatment of different types of coal. Basically there are four types of coal found in nature. These are bituminous, sub-bituminous, anthracite and lignite. Each type of coal is chemically distinct with others and varies significantly with respect to its calorific value, chemical configuration, amount of ash present, and in geographical origin. Oxides of silicon, aluminum, and iron are the chief components of bituminous coal. Variable amount of unburnt carbon present can be measured by the LOI which is one of the most significant chemical properties of FA. The ash generated from lignite coal is rich in Ca and Mg oxides typically ranges between 12-25% with decreased amount of silica and oxides of iron ( $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ), as compared to bituminous coal FA [24]. Very little amount of carbon can also be found in Lignite coal FA. Apart from the handling condition of ash i.e. either wet or dry there is several more classification out of which the ash coming out from coal is one. Anthracite coal FA finds little usage due to the large amount of carbon present in it. Only some amount of Anthracite coal is combusted in utility boilers, thus generating low concentration of FA. Moreover Lignite and subbituminous coal FA possesses greater concentration of sulfate ions. The acidity and alkalinity of FA can be projected on the basis of amount of Sulphur, calcium, and magnesium present in the parent coal. During combustion of coal high amount of Sulphur is generated which in turn characterizes FA as acidic in nature. While on the other hand if the Sulphur content of parent coal is low then the FA produced from such coal will result it in more alkaline. FA matrixes contain salts of hydroxide and carbonate on the substrate of calcium and magnesium which shows their alkaline behavior of ash residues [25]. Consequently, the Potential to hydrogen (pH) of fly ash particles lies between the limit of 4.3 to 12.5 [26]. The electrical conductivity of Fly ash particles is in the order of 0.63 to 55  $\text{ds m}^{-1}$  due to the existence of soluble salts [25]. The reactivity of lime normally increases with increase in silica content of the FA. K.C.Sahu studied the leaching characteristics of fly ash in which Ash ponds faces an extreme problem with leaching of heavy dead weight metals. Hence in order to overcome from such issue a fixed quantity of Ash is mixed with water at required pH. The period of time may be fixed or variable. The whole process seems to be decreasing convergently with time.

## 2.4 Uses of Fly ash

Fly ash finds application in various sectors. In broad, the use of FA can be band together in to three categories.

- Huge amount of FA is utilized in various areas which are of little significance in terms of cost. Some of the common uses are in brick industry, filling of mines, ridges, surfacing and recovery of fallow land etc. A lot of efforts have been made for manufacturing of bricks by different agencies using lime, different types of resins, gypsum and clay. These binders are mixed with FA at different ratio and a range of products are available in the marketplace. Bricks made of FA are more highlighted since it saves the valuable top layer of soil. Similarly, exhaustion of river sands for mine stowing in underground collieries would lead to use of ash in large scale for filling up of mine excavations. The study of Leaching action with FA is essentially required before use.
- FA are commonly used as cement stabilizer, light weight filler materials for prestressed structures ,wall slates and roofing tiles ,for insulating blocks ,in paints and enamels and as herbicide in agricultural science to destroy unwanted vegetation. These all comes under the category of medium cost values.
- Recovery of various Magnetic oxides, Aluminum oxides ( $\text{Al}_2\text{O}_3$ ), and different trace elements , synthesis of Zeolites for industrial applications and making of inorganic wools. Removal of bleaches and organic compounds from waste water, mercury from the flue gases, adsorbents for cleaning of flue gases ( $\text{SO}_x$  and  $\text{NO}_x$  emissions). These are grouped under high cost values.

## 2.5 FA disposal –curse to environment

Large amount of solid wastes in the form of Fly ash have been generated from thermal power plants. These wastes are widely utilized in various construction materials and other sectors. Apart from fulfilling the needs, the disposal of FA is the burning problem and creates hindrance in developing a pollution free nation. Hence it's a matter of great concern. Some of the problem regarding FA disposal are mentioned below:-[27]

- Fly ash particles are available in both the dry and wet state. These ashes are disposed in bulk which occupies thousands of hectares of land and destroys the fertility of top soil.
- Handling of FA particles in dry condition is a tough job. Since these ashes are very fine and dispersive in nature. The fine tiny bits of FA destroy the structural shells and affects cultivation.
- It hampers the ecosystem through various modes of pollution i.e. soil, air and water.
- Since FA are disposed in open atmosphere which ultimately results in various air borne diseases due to long intake of air.
- The biological features of soil and overall yield of crops also get hampered when FA is disposed in the nearby areas before any treatment.

## 2.6 Reviews on FA

A lot of investigators have worked on the coal ash properties to evaluate its importance in various fields. Some of them are mentioned below:-

**Sherwood and Ryley** stated that Fly ash possesses self-hardening features due to the presence of free lime in the form of calcium oxide or calcium hydroxide.

**McLaren and Digioiab** presented that the specific gravity of Fly ash is relatively lower than that of soils. The density of the ash fills gets reduced which is a major advantage in terms of its use as various filler materials. Now these fillers can be used in spongy walls and ridges particularly when the foundation is weak.

**Sridharan et al.**, studies the micrographs of FA particles through SEM. These particles are mostly solid spheres with glassy appearance, hollow spheres with smooth-edged porous grains, asymmetrical agglomerates and irregular absorbent scraps of unburnt carbon. Presence of iron particles which are dark grey in color can be identified as pointed grains.

According to **Mohini Saxena and P.Asokan** a lot of multidisciplinary tests on coal ash have been conducted at various lab centers. Regional Research laboratory, Bhopal has worked a lot on FA and enhanced the various methodologies for pilot scale demonstration. They cultivated Crops,



vegetables and cereals and reported that the yield increases greater than before by FA utilization with no toxicity. They also developed paints using FA and epoxy systems for safety and embellishment. These FA paints has improved resistance to rust, abrasion and wear.

**Mitchell and Brown** said that the soil, FA and lime displays unique behavior and are much more dependent on the physicochemical properties of the fly ash and soil like porosity, segregation ,lime content ,time and pressure applied during compaction.

**Sevelius et al.**, studied the utilization of Fly ash and Bottom ash in bricks manufacture and in refractory products. He also studied that there is a remarkable increase in the consumption of FA as a basic raw material.

**Mathur and coworkers** focused on the influence of heavy weight metal present in FA on various species of plant like Ipomeas carnea, Typha Angustata and calotropis procera.

**Martinet al.** stated that FA in wet but unsaturated state displays cohesive properties which are due to the tensile stress developed by the capillary action of water. Since this property limits the long term solidity of the compacts. He concluded that for improving the mechanical strength angle of shearing is more important.

**Indraratna et al.** showed a comparison between the intercept of cohesion and angle of shearing resistance of dry and wet fly ash specimens .He reported that there is 100% loss of cohesion mainly to dry specimen with no change in resistant shearing angle.

**Gray and Lin** studied the difference in specific gravities of the Fly ash and displayed that the differences are due to the particle shape (sphere, plerospheres, etc.), and chemical configuration.

**Rajasekhar** specified that fly ash particles are mostly amorphous (glassy) with spherical shape. The low specific gravity is due to the existence of large number of small hollow spheres enclosed in big spheres (plerospheres). Reason behind that the trapped air cannot be detached from hollow spheres or due to the difference in configuration of these particles.

**Singh and Panda** determined shear strengths of newly made Fly ash compacts in the presence of different volume of water content. He concluded that the shearing strength of the compacts is due to the internal friction.

**Molliner AM & Street JJ** stated that in agriculture, FA can be used either in the form of an amendant or manure without any alteration in the chemical configuration. A lot of vegetables and crops displayed enhanced acceptance of various elements like B, Ca, Fe, Hg, As, Mg etc. when they grew under the condition of 10% acidic ash .These elements contribute a lot in the variation of crop yield and thus the uptake is directly relational to used ash .FA has been known to increase crop yield by nullifying soil acidity and alkalinity.

**Lee H, Lee CH, and Kim PJ** stated that Fly ash when incorporated with soil improves its physic-chemical and organic properties which has its great benefit in agricultural sector. Many agricultural scientists have proved that FA possesses almost all the essential nutrients which are required for cultivation of crops. However it is not used as commercial manure.

## 2.7 Reviews on Fly Ash Bricks

Fly ash based bricks offer exciting advantages over traditional clay bricks. Aggressive research is being carried out worldwide on fly ash based geo polymers to improve functional properties. This chapter outlines some of the recent reports published in literature on fly ash based geo polymers, its utilization for making bricks and its mechanical properties. Fly ash bricks have created prodigious attention and awareness among materials experts and engineers in current years due to the considerations of developing an environmental friendly, high strength material and partially switching currently used clay bricks.

**Obada Kayali** studied the properties of Fly ash and clay made bricks and concluded that the mechanical properties of fly ash bricks have exceeded to those of standard load bearing clay bricks. Compressive strength was 24% better than good quality clay bricks and tensile strength was nearly three times the value for standard clay bricks. The bond strength of the fly ash bricks is 44% higher than the normal clay bricks. The density of fly ash bricks is 28% less than that of standard clay

brick. The reduction in a weight of bricks results in a great deal of savings in terms of raw materials and transportation costs. Fly ash brick can easily soak up mercury from normal air which is in contact with it and thus makes it cleaner for berating. There is also a process named carbonation in which fly ash absorbs carbon dioxide from normal atmosphere due to which carbon sequestration occurs and the amount of carbon gets reduced in atmospheric air which helps in minimizing global warming.

**Sunil Kumar** has presented an extensive review in reported work on fly ash bricks. He investigated the flexural strength, water absorption test, density, porosity and stability of these solid bricks and hollow blocks. He witnessed that these bricks and blocks have enough strength for their usage in low rate housing growth. Tests were conducted to determine the compressive strength and hardening effects and to analyze the effects of curing with time. The compacts treated in hot water shows better strength and hardening effects as compared to normal water cured compacts. Initially the strength of these blocks and bricks increases with higher rate and then at a comparatively lower rate. There is a direct relationship between FA and water absorption. As the content of FA increases water absorption also increases .And on the other hand water absorption decreases with increase in density of the FA compacts. These FA bricks and blocks with proper phosphogypsum extent have improved resistance to robust sulfate environment.

**Ball MC & Carroll RA** has studied the various bricks manufacturing methods and understands the reason behind the strengthening effects of these autoclaved FA bricks. The FA bricks becomes hard mainly due to the formation of calcium silicate hydrate and calcium aluminate silicate hydrate .The hydrothermal reaction takes place between silica ,alumina and water when the compacts are allowed to cure under the steam bath normally at 110<sup>0</sup>C -180<sup>0</sup> C. The presence of Tobermorite phase also helps in enhancing the hardenability of the Fly ash bricks.

**Cultrone G, Sebastian E, and Elert K**, studied the permeability of FA bricks and co-relates its effects on various chemical and mineralogical configuration of Fly ash particles. FA bricks also depend on the temperature of firing resulting in to more vitrified dense structure and phenomenal change in shape and size.

**Dimitrios Panias and Ioanna P.** stated that in order to develop the better mechanical strength, water content has displayed as a critical parameter in the synthesis of FA built geopolymers. Water displays its importance during suspension, poly condensation and the different hardening junctures of geo polymerization. The presence of NaOH badly affects the compressive strengths during formation of geopolymers. The Geopolymers which are synthesized either in higher or lower NaOH content (aqueous phase), results in reduced compressive strength. The concentration of Sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) solution in the formation of geopolymers showed a significant effect on the acquired compressive strength.  $\text{Na}_2\text{SiO}_3$  solution controls the solvable silicate gathering and the major silicate classes in a geopolymeric system, thus increases the strength of the formed materials.

**Mridul Garg, Manjit Singh and Rakesh Kumar** have studied the durability of the FA, phaspho gypsum and lime based binder and its execution in water along with accelerated ageing. The cementing binder that are cured at  $50^\circ\text{C}$  displays little porosity with improved water resistance as compared to that of  $27^\circ\text{C}$  cured binder. The  $50^\circ\text{C}$  cured binder displays little reduction in strength and mass with rise in temperature and in alternate moist and dried cycles.  $50^\circ\text{C}$  treated binder displays no drop in strength and mass from the pristine standards, with rise in temperature from  $27^\circ\text{C}$  to  $50^\circ\text{C}$  and with different heating and freezing sets. Hence these are ideally suited for making structural blocks, slates, panels etc.

A blue geometric graphic consisting of several overlapping triangles and quadrilaterals, creating a dynamic, angular shape that points towards the right.

## CHAPTER 3

# Experimental Work and Methodology

### **3. Experimental work and methodology**

#### **3.1 Introduction**

Fly ash has been used in various architectural and industrial applications on large scale. Hence Consumption of this huge amount of fly ash greatly reduces the difficulties met by coal based TPPs for its dumping. Analysis on the performance of FA at various states is essentially required before its usage. So to understand the characteristics features of FA, experiments cannot be performed on field domain. There is no any alternate option except research laboratory test to assess its importance. The research conducted in laboratory provides a calculative approach to govern several parameters that come across during practice.

Brief description of the types of material used, sample preparation and its characterization through SEM, XRD, and FTIR, Mechanical and surface properties like Compressive strength, Hardness and wear resistance, Thermal conductivity measurement and others are outlined in this section.

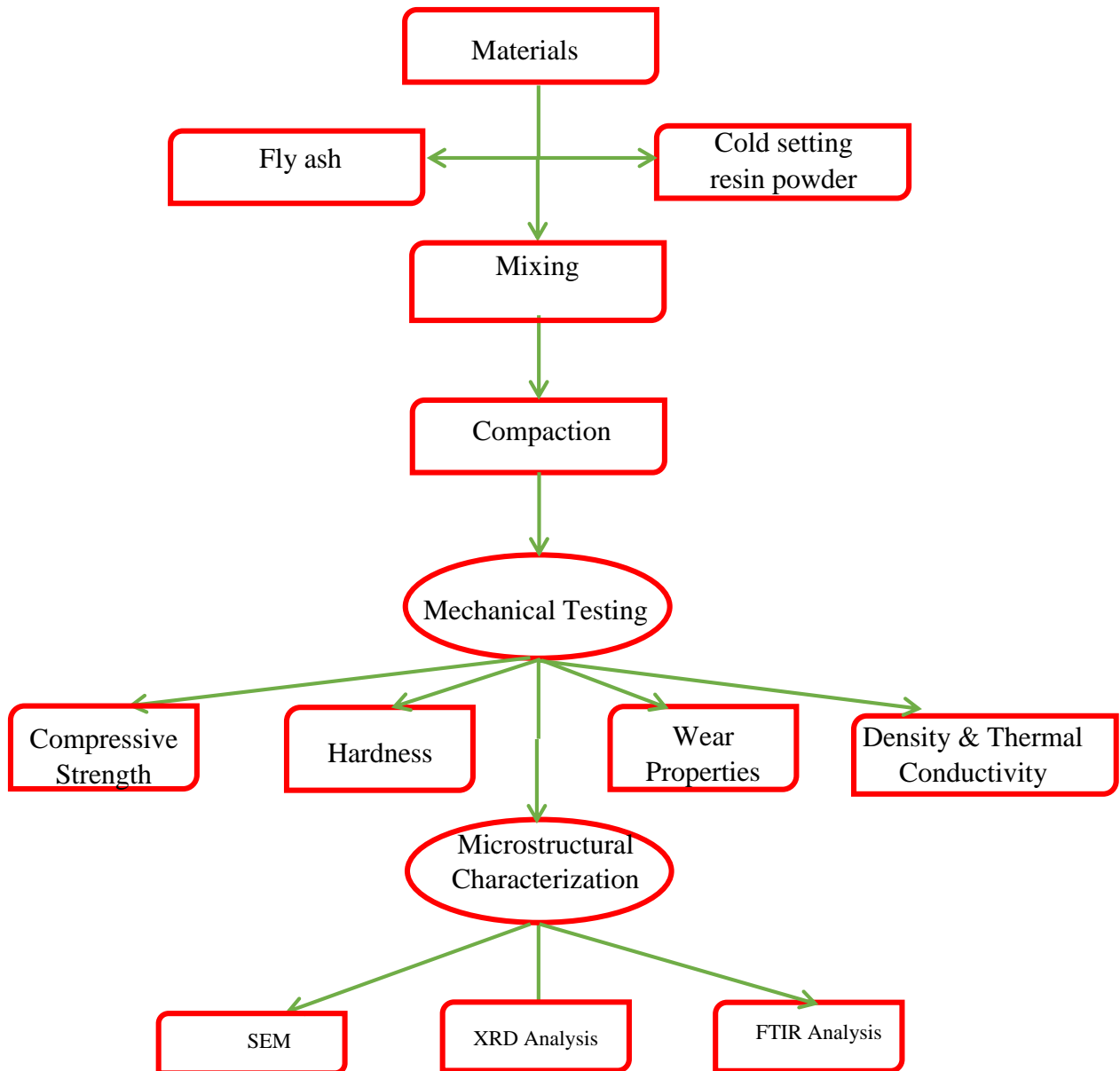
#### **3.2 MATERIALS USED**

##### **3.2.1 Fly ash**

The Fly ash used in this project was collected from electrostatic precipitators of the captive power plant (CPP-II) in dry condition. The fine powders were oven dried at 110°C-160°C and kept in air tight bottle for later use.

##### **3.2.2 Cold setting Resin and Binder**

The resin powder and hardener used in the present study was supplied by Geosyn private Ltd. Kolkata.

**Flow chart of experimental procedure**

### **3.3 Experimental Methods**

**3.3.1 Preparation of Samples:** The samples were prepared by Powder metallurgy route.

#### **3.3.1.1 Mixing**

Three different weight percentages of Fly ash and resin powder with (75%, 80% and 85%) and (25%, 20% and 15%) were taken respectively. These compositions were mixed thoroughly by a mechanical vibrator (Abrasion Tester Model PEI-300), to get a homogenous mixture. Different compositions of Fly ash along with resin powder were kept in three different small size bottles. Around 6-10 small steels balls are kept inside for proper mixing. Mixing was done till the vibrator shows 1000 revolutions which almost took five hours.

#### **3.3.1.2 Compaction**

The compaction experiments were executed to make cylindrical FA compacts. Cylindrical die and punch having 15 mm diameter made of stainless steel was used to make cylindrical Fly ash compacts. Mixture of approximately 5 gm. was taken for each composition. Then the punch & die was cleaned with cotton followed by acetone so that all the dust is removed from the inside surface of the die and outside surface of the punch. Then greasing was done to avoid sticking. The mixture prepared earlier was poured inside carefully. During the packing slight shaking was done to accommodate the maximum possible amount of material. Finally the whole system was subjected to hydraulic seal valve made tight, mounting was done coaxially. Maximum of 6tons of load was applied on it very slowly. Once the maximum load was achieved, the apparatus was powered off. The whole system was relaxed for 5 minutes which then followed by unloading. Compact was ejected from the Die in the same direction as the compression and was kept in normal atmosphere for 1 day. The cold setting liquid (hardener) was applied on the surface of the compacted samples with the help of a dropper, so as to harden the newly made compacts. The amount of Hardener used was  $1/6^{\text{th}}$  or  $1/4^{\text{th}}$  of the mixture. Hence in this way twelve samples for each composition were made. All the samples were dried in open atmosphere for 2 days.

#### **3.3.2 Water treatment**

Three samples from each composition were cured in water at 110°C -180 °C for 48 hours.



### 3.4 Determination of Mechanical properties

#### 3.4.1 Hardness

Vickers hardness tester (LECO, LM 248AT) as shown in Figure 3.1 ,was used to find the hardness values of all the dry and wet samples using 20 gf Load for a dwell time of 15 seconds. At least eight measurements were taken at different position for each sample in order to get constant results.



Fig.3.1- Leco, LM 248AT Micro indentation Hardness Tester

#### 3.4.2 Compressive Strength

In order to measure the compressive strength of dry and wet samples INSTRON 1196. Prior to test, gauge length and gauge diameter of the dry and wet specimens were measured individually by the aid of Vernier caliper .The tests were carried out at room temperature (300 K) with a constant crosshead speed of 1mm/min and the full scale range load of 50 kN. This computer

integrated machine gives the Load vs displacement signals directly when the specimens were subjected to tests.

### 3.4.3 Wear resistance and Friction

In this study computerized Ball on Plate Wear Tester (TR-208-M1) as shown in Figure -3.2 was used to evaluate the wear performance and sliding contact resistance of the Fly ash compacts. The experiment was carried out with the help of 4 mm diamond indenter keeping the different track radius of 4 and 8 mm respectively. Prior to wear, constant normal load of 10 and 20N was applied. The indenter rotates on fly ash compact with a constant speed of 20 rpm for different time period of 600s. At the end of each test, loss in weight of the samples was noted. Results obtained have been expressed in terms of wear depth, and friction co-efficient.



Fig.3.2- Ball-On-Plate Wear Tester (TR-208 M1)

### 3.4.4 Thermal conductivity

To measure the thermal conductivity of Fly ash and resin powder mixture, KD2 Pro analyzer as shown in figure 3.3 was used and it follows ASTM Standard D5334-08 [28]. It comprises of a handheld controller and a various sensors that operator can embed into very nearly any material. Single probe of 6cm long and 0.127 mm diameter was inserted in a small plastic bottle filled with FA & resin powder to find the conductivity value .At least ten values of each composition was

recorded to get the appropriate result. KD2 Pro uses the transient line heat source mechanism to evaluate the conductivity and diffusivity of the given mixture. A restrictive calculation fits time and temperature information with exponential functions via nonlinear least squares technique.



Fig. 3.3 KD2 Pro analyzer

### 3.4.5 Water Absorption

The cylindrical compacts were tested for water absorption according to ASTM C642. The weights of all the samples were taken. The compacts were first dried in an oven at 100<sup>0</sup>C -120 <sup>0</sup>C ensuring removal of moisture and hence allowed it to cool at room temperature. The weights were taken after drying and the variation in weight was less than 5%, considered it as dry. Now the compacts of different composition was immersed in a beaker filled with water and was kept in an oven at 110<sup>0</sup>C-180<sup>0</sup>C for 48 hours. Compacts were surface dried after removal and final weight was measured. The amount of water absorbed (%) was calculated using equation-1.

$$\frac{M2 - M1}{M1} * 100 \text{ ----- (1)}$$

Where;

M<sub>1</sub> and M<sub>2</sub> are the mass of dry and wet sample respectively.

### 3.4.6 Density

On the basis of Water absorption test, the density of dry and wet compacts was calculated.

## 3.5 Microstructural Characterization

### 3.5.1 SEM Study

In present study, A JEOL 6480 LV Scanning Electron Microscope (Fig. 3.4) was used for the characterization of microstructural changes (pits, cavities, and porosity), determination of particle size and morphology of FA compacts. To get the better image resolution, secondary electron imaging with accelerating voltage of 15 KV was used.



Fig. 3.4 Scanning Electron Microscopy (JEOL JSM-6480LV)

### 3.5.2 XRD Study

The mineralogical composition of Fly ash and the different phases present was determined by XRD analysis in a Philips X-pert multipurpose x-ray diffractometer (shown in figure.3.5) using  $\text{Cu K}\alpha$  ( $\lambda=1.5418\text{\AA}$ ) radiation. The patterns were examined by comparing the positions of peak and intensities of the samples with those in the (JCPDS) data files. The diffraction patterns were recorded in the scanning range of  $20^\circ$ - $80^\circ$  with a step size of  $2^\circ$  C per minute.



Fig.3.5 Philips X-pert multipurpose x-ray diffractometer

### 3.5.3 FTIR Study

FTIR spectroscopic technique is used to understand the chemistry of surface for fly ash in thermally active state along with different state of mineral phases,  $\text{H}_2\text{O}$  and  $-\text{OH}$  group on silica and alumina. Fourier transforms infrared radiation (FIR) spectrometer (shown in figure.3.8) is used to calculate the transmission percentage of infrared. In order to prepare pellet little quantity of potassium bromide (KBr) was segregated with powder sample and after that pressing of mixture was done. Analysis of that pellet was done using FIR by keeping the pellet in sample holder.



Fig.3.6 Perkin-Elmer Spectrum RXI, (FTIR) Spectrometer

A blue geometric graphic consisting of several overlapping triangles and quadrilaterals, creating a dynamic, abstract shape that points towards the right.

## CHAPTER 4

# Results and Discussion

## 4. Results and discussion

### 4.1 Composition of Fly ash

FA mainly consists Silica ( $\text{SiO}_2$ ), Alumina ( $\text{Al}_2\text{O}_3$ ), Calcium Oxide ( $\text{CaO}$ ), and Iron Oxide ( $\text{Fe}_2\text{O}_3$ ). The chemical composition of Fly ash is tabulated in table 4.1.

Compounds	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{CaO}$	Mgo	$\text{P}_2\text{O}_5$	$\text{Fe}_2\text{O}_3$	$\text{SO}_3$	$\text{K}_2\text{O}$	LOI
Composition (%)	54.5	26.5	2.1	0.57	0.6	-	-	-	14.18

Table 4.1 Compositional analysis of Fly ash

### 4.2 Water Absorption Test

Table 4.2 shows the amount of water absorbed corresponding to different FA composition. The water absorption values of FA composites lies in the range of 15.55 % to 19.09%. It can be seen that all the composition met the absorption criteria set by several developing countries. India permits the maximum of 20 % water absorption when compacts are immersed for 24 hours.

Mix Composition (Wt. %)	Weight (gm)		Water Absorption (%)	Average Water Absorption Value (%)
	Dry	Wet		
(FA) <sub>75%</sub> + (RP) <sub>25%</sub>	4.579	5.302	15.78	15.55
	4.630	5.340	15.33	
(FA) <sub>80%</sub> + (RP) <sub>20%</sub>	4.452	5.151	15.70	16.61
	4.642	5.456	17.53	
(FA) <sub>85%</sub> + (RP) <sub>15%</sub>	4.502	5.356	18.96	19.09
	4.329	5.162	19.23	

Table 4.2 Percentage (%) water absorbed by various FA polymer compacts



Fig.4.1 shows a relation between the amount of water absorbed and density of dry composite with respect to FA composition. It is evident from the graph that the water absorption increases with increase in FA content. 85wt. % FA absorbs water to a maximum of 19.09%. This indicates that that most of the openings of the compacts are open to outside.

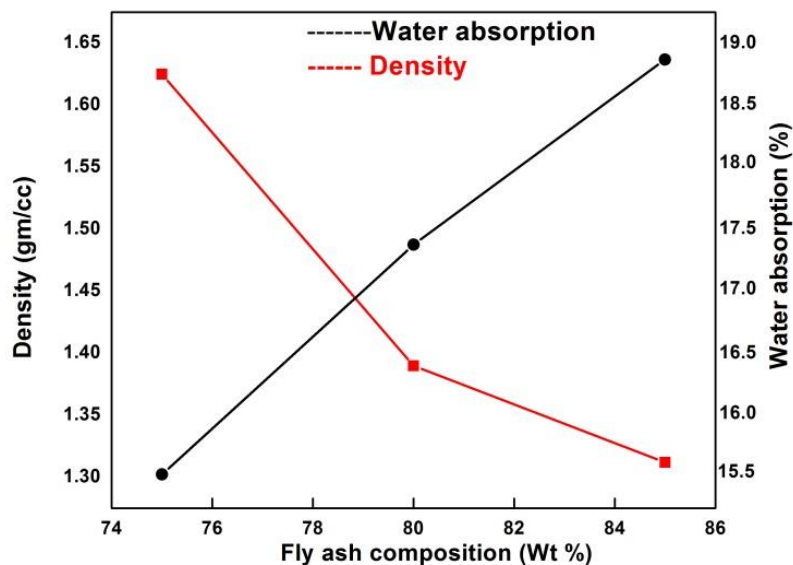


Figure.4.1 Water absorption and density as a function of FA Composition

### 4.3 Density Measurement

Density of the samples was calculated before and after treatment. From Fig. 4.2 we can say that density of dry compacts decreases with increase in weight percentage of FA. As the dry compacts are immersed in water at 110<sup>0</sup>C -180<sup>0</sup>C, then through capillary action voids are filled and it becomes hard and the porosity is eliminated. As a result of which the compacts become dense and finally the density increases with increase in FA content.

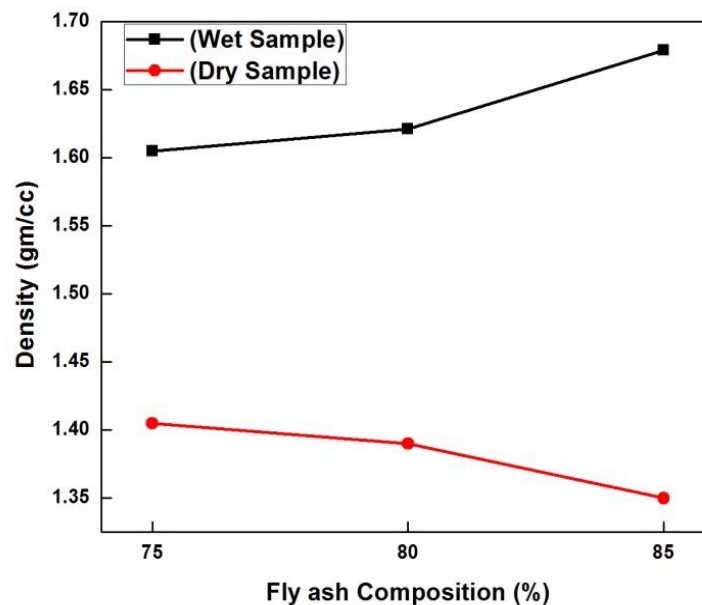


Fig.4.2 Variation in dry and wet density w.r.t FA composition

Mix Composition (Wt. %)	Density (g/cm <sup>3</sup> )	
	Dry	Wet
(FA) <sub>75%</sub> + (RP) <sub>25%</sub>	1.40	1.60
(FA) <sub>80%</sub> + (RP) <sub>20%</sub>	1.38	1.62
(FA) <sub>85%</sub> + (RP) <sub>15%</sub>	1.35	1.67

Table 4.3 Density value of dry and wet FA polymer compacts

#### 4.4 Hardness Measurement

Hardness values of all the Fly ash polymer composite of different compositions, both in dry and wet state, were measured by the help of LECO, LM 248AT Vickers hardness tester. The Hardness values as obtained are shown in Table 4.4. The values of hardness are in the range of 32.93 HV – 44.08 HV for dry composites and 39.78 HV – 47.37 HV for wet FA composites respectively.

S.NO	Mix Composition (Wt. %)	Micro hardness value (HV)	
		Dry	Wet
1	(FA) <sub>75%</sub> + (RP) <sub>25%</sub>	32.93	39.78
2	(FA) <sub>80%</sub> + (RP) <sub>20%</sub>	38.26	43.04
3	(FA) <sub>85%</sub> + (RP) <sub>15%</sub>	44.08	47.37

Table 4.4 Hardness values of various FA resin mix compacts

Fig. 4.3 shows a comparison between the hardness values of dry and wet fly ash composites. It is evident from figure that as we go on increasing the wt. % of FA, i.e., resin content decreases the hardness values of both the wet and dry compacts increases. Maximum hardness values in case of 85 wt. % FA is achieved.

It is evident from the XRD analysis as shown in fig. 4.4 (b) of water treated 85 wt. % FA compact that a Calcium Silicate Hydrate (C-S-H) and Calcium Aluminate Silicate Hydrate (C-A-S-H) phase appears which are responsible for the hardness improvement. Both these phases are formed by the reaction of  $\text{Ca}(\text{OH})_2$ ,  $\text{SiO}_2$  and  $\text{H}_2\text{O}$  when treated in water at  $110^\circ\text{C}$ - $180^\circ\text{C}$ .

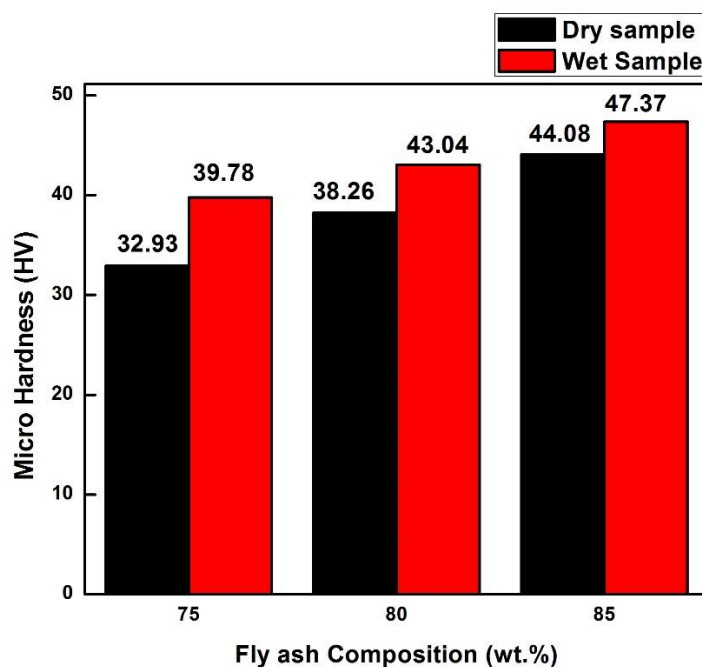


Fig. 4.3 Variation in hardness values with wt. % of FA

## 4.5 XRD Analysis

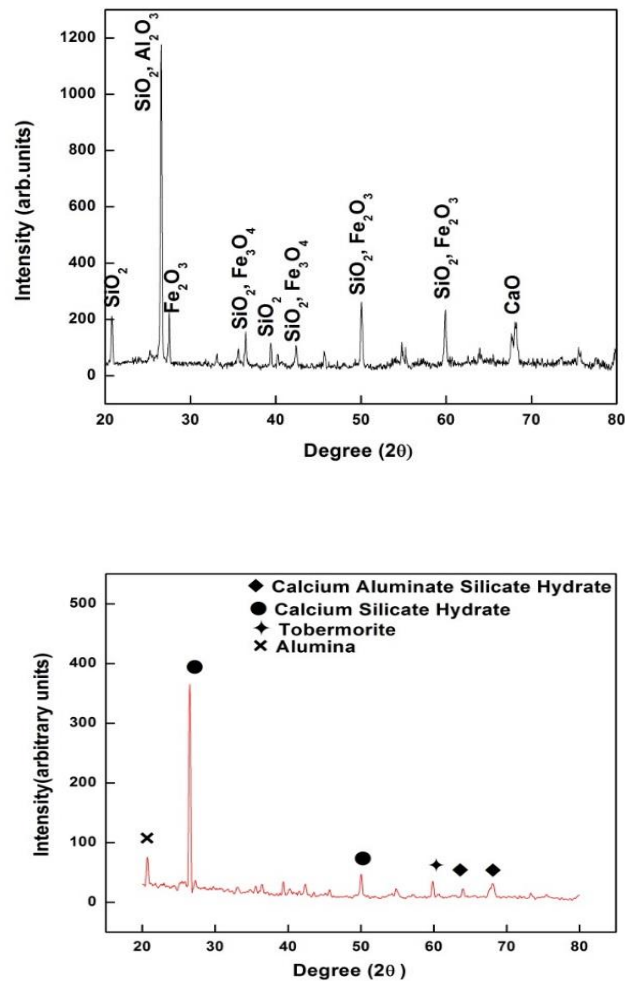


Fig.4.4 (a) XRD analysis of Fly ash and Fig.4.4 (b) XRD analysis of water cured compact

Fig.4.4 (a) shows that Fly ash particles primarily consists of Silica and Alumina. Fig 4.4 (b) Shows the XRD analysis of water treated compacts. It has been found that in the presence of moisture, pozzolanic reaction occurs that leads to the formation of new phase i.e. calcium silicate hydrate (CSH) and calcium aluminate silicate hydrate (CASH) .These phases are responsible for solidification of unfired compacts and hence creating strong structures, excellent inter particle

bonding with improved mechanical properties like hardness etc. CSH and CASH are considered to be an initial reaction product which changes in to a semi crystalline solid phase called Tobermorite ( $C_5S_6H_5$ ).

#### 4.6 FTIR Analysis

Fig: 4.5 shows the Fourier transforms infrared radiation (FTIR) spectrometer plot of 100 % FA along with 80% FA + 20 % RP mix . It can be seen that for 80 % FA mix the (%) transmittance is getting decreased with respect to 100% FA .With comparison of FTIR spectrum phase transformation of FA and FA mix can be recognized. The most characteristic difference between the FTIR spectrums of these two is the shifting of band attributed to the asymmetric vibrations of Si-O-Si and Al-O-Si. The broadness in band appeared to be around  $1250\text{ cm}^{-1}$  in the FTIR spectrum, which became sharper as compared to FA mix. . Then after these bands starts shifting towards low frequencies at around ( $950\text{ cm}^{-1}$ ) indicating the formation of a gel like phase named alumino silicate which is connected with the suspension of fly ash in the strong alkaline activating solutions. Stretching vibration of Si-O-Al appeared at around  $600\text{ cm}^{-1}$ . The wide band groups showed up in both IR spectra in the area of  $3500\text{ cm}^{-1}$  are assigned to stretching (-OH) and bending (H-O-H) vibrations of bound water atoms, which are surface consumed or entangled in the huge depressions of the polymeric skeleton [30, 31]. This broadness indicates the presence of strong hydrogen bonding [32].

As a conclusion, water content is a crucial synthesis parameter that affects their mechanical strength. Peaks appeared around  $2400\text{ cm}^{-1}$  attributed to O-H stretching. The gradual decrement in the intensity and broadness in the band confirms the loss of water. Peak at  $3000\text{ cm}^{-1} - 2000\text{ cm}^{-1}$  could be assigned to C-H stretching vibration of organic contaminants which may be introduced during sample handling or some hydrocarbon present in fly ash [33].

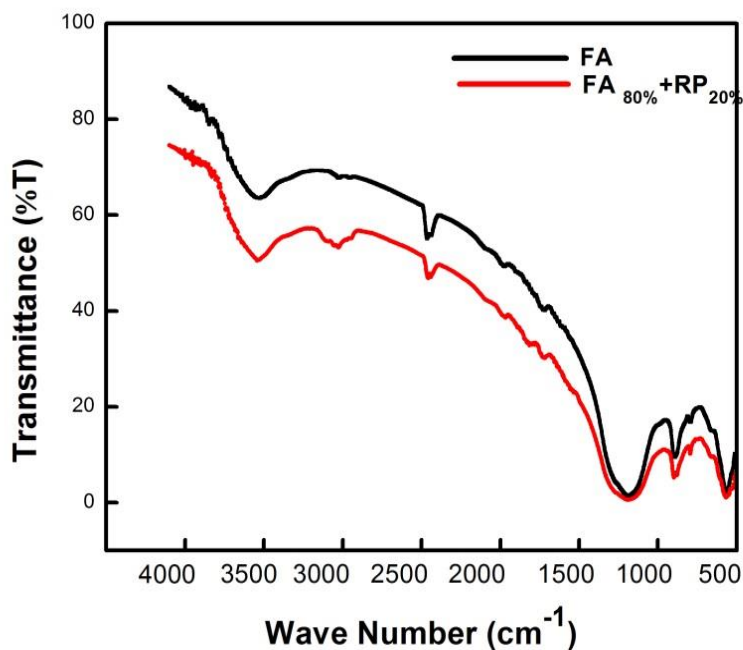


Fig.4.5 IR spectra of the FA and FA resin Powder mix

#### 4.7 Determination of Compressive Strength

The compressive strength measurement of the cylindrical samples was done as per standard practiced. Test was conducted on the three samples of each composition and the average value of all is evaluated. Table 4.5 shows the strength values of different compositions of FA, both in dry and wet state. For dry composites, the Compressive strength value lies in the range of 6.5 to 11.28 MPa. 85 wt. % FA compositions have got the highest strength value while the lowest strength value of 6.5 MPa was gained by 75 wt. % FA composition.

S.NO	Mix Composition (Wt. %)	Compressive Strength (MPa)	
		Dry	Wet
1	(FA) <sub>75%</sub> + (RP) <sub>25%</sub>	6.5	5.52
2	(FA) <sub>80%</sub> + (RP) <sub>20%</sub>	8.73	7.98
3	(FA) <sub>85%</sub> + (RP) <sub>15%</sub>	11.28	9.43

Table 4.5 Compressive strength values of different FA resin mix compacts

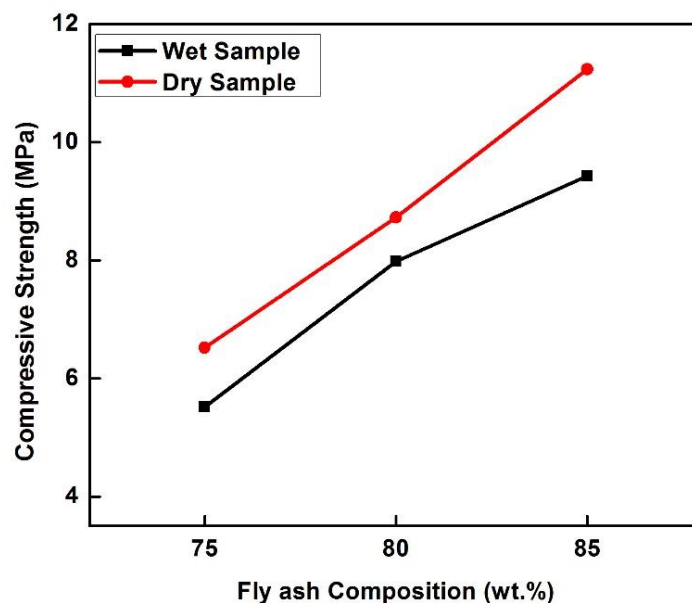


Fig.4.6 Compressive Strength of Compacts at different FA compositions

It can be seen from Figure.4.6 that the composition of (FA)<sub>85%</sub> + (RP)<sub>15%</sub> has higher compressive strength than other two compositions. It is found that with decrease in the resin percent with fly ash mix has increased the compressive strength. As it is evident from SEM micrographs that 75 wt. % FA mix composite possesses cracks which leads to decrement in compressive strength. As the percentage of FA is increased there is a good bonding between the interfaces which leads to improvement in strength of the compacts. These observations confirm that addition of cold setting resin powder in excess to fly ash may not be beneficial. Here resin powder is only used as a binding agent. Water treatment shows a little bit negative impact on the strength of composite.

#### 4.8 Thermal Conductivity measurement

Thermal Conductivity is the property of a material depicting its capacity to exchange heat. The Thermal Conductivity of the Fly ash –Resin Powder mix were determined through Hot wire method using KD2 pro analyzer. It was reported that the conductivity of FA-resin powder mix decreases with increase in FA content. 75 wt. % of FA composition displayed the maximum

thermal conductivity value with an average of 0.0552 W/mK. Only FA powder shows a conductivity value higher than other three compositions.

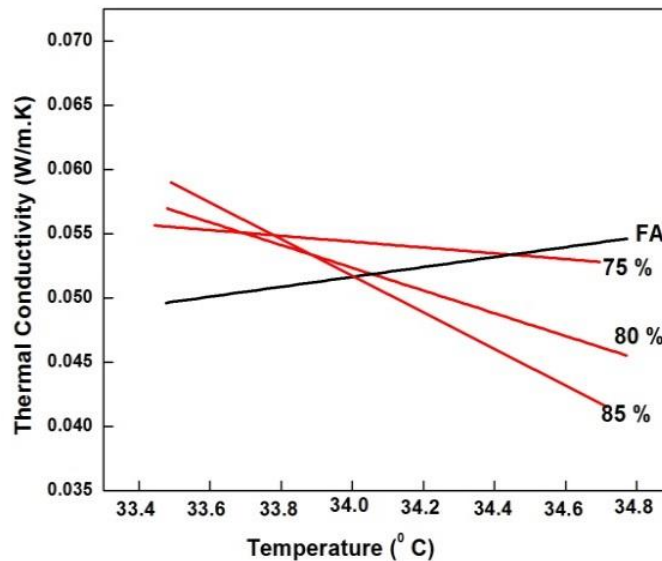


Fig.4.7 Thermal conductivity of FA –Resin powder Mix at different compositions

The thermal conductivity of clay is around 0.82 W/mK which is very much higher than FA. Hence it can be concluded that FA can be used as a substitute material instead of clay. An important result can be drawn from the graph that by adding the resin powder, conductivity value decreases to some extent. The low thermal conductivity of FA is desirable for making several electronic insulating devices. Tests were carried out to assess the insulation capability of the resin added FA mix, shown in Fig 4.7. It is found that the thermal conductivity of FA increases with increase in temperature, whereas in case of resin powder FA mixes, the conductivity of composite decreases with increase in temperature.



## 4.9 Wear resistance and Friction study

### 4.9.1 Wear Study

Wear characteristics of FA polymer composites were carried out at different loads of 10N and 20N. Fig.4.8 (a) shows the plot between wear depth ( $\mu\text{m}$ ) and time (s) of dry composite at load of 10N. It can be seen that the wear resistance of 75 wt. % of FA composition is less as compared to the other two composites. It can also be correlated from the results of hardness value mentioned in Table 4.4. For dry compacts, 75 wt. % FA possesses less hardness value than the other two. Similar trend in wear behavior was observed in both the cases. Fig.4.8 (b) shows a plot between wear depth ( $\mu\text{m}$ ) and time (s) of wet compacts at 10N load. In this case too, 75 wt. % FA compact is less resistant than other two. Since the hardness value of this is lower than the other two compositions. The only difference is that the wet compacts became harder in presence of moisture which in turn the wear depth decreases to a value of (275-250  $\mu\text{m}$ ) and is lower than dry compacts (350-280  $\mu\text{m}$ ).

Fig.4.8 (c) and (d) shows the plot between wear depth ( $\mu\text{m}$ ) and time (s) of dry and wet compacts at load of 20 N respectively. From figure (c), it can be seen that by applying load of 20 N the wear depth increases to a value of 350-450  $\mu\text{m}$  which is comparatively higher than that of 10 N weared dry compacts.

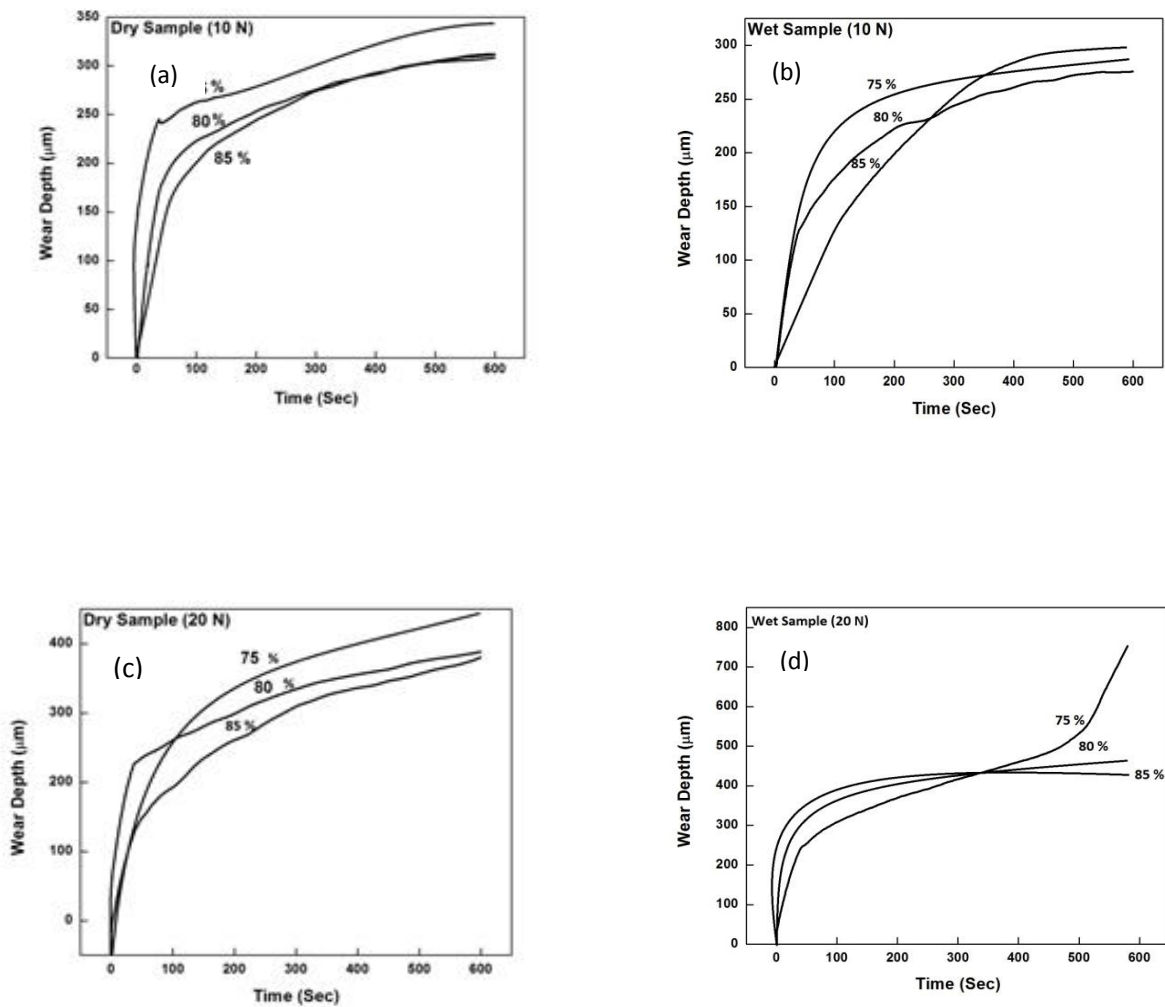


Figure 4.8 Wear behavior of FA compacts at different loads

Since wet compacts are very much harder than the dry one hence by the application of 20 N load smooth curves appears as shown in fig. (d). The wear depth value in this case decreases little bit and this curve follows the trend of steady state. It can also be observed that for the 20 N applied load at a time of about 300 sec the wear depth becomes same irrespective of FA composition. At first it reaches to a value of 400 μm and then it follows constant horizontal saturated line. Moreover wear behavior can also be co-related with the help of wet density.

Figure 4.9 (a & b) shows the variation in material loss (gm) vs time (sec). It is found that material loss decreases with increase in sliding time implies the reduction in wear rate with time. The weight loss is more in case of dry samples than water treated samples. There is not much variation with applied load.

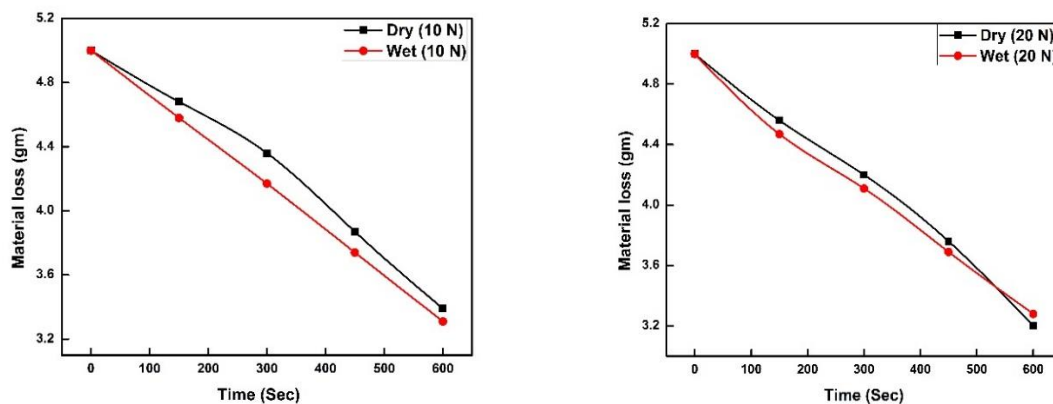


Fig.4.9 (a & b) shows the variation in weight loss (gm) with respect to time (sec).

### 4.9.2 Friction Study

The frictional behavior of Fly ash polymer composites has been shown in Fig.4.10. The average co-efficient of friction ( $\mu$ ) value of all the composites has been reported to be (1.1-1.4). Chapman et.al, Suggested that spines in the frictional behavior of the composites could be related with the flaw generation from the edges of the material. They estimated that spines in the value were accompanied with an elevated pitched noise [29].

From our study as shown in Figure 4.10 (a & b), it is observed that initially there is a slight increase of co-efficient of friction ( $\mu$ ), but just after few seconds the  $\mu$  value follows linear trend throughout the further time of testing. It can also be seen that co-efficient of friction decreases with an increase in FA composition.

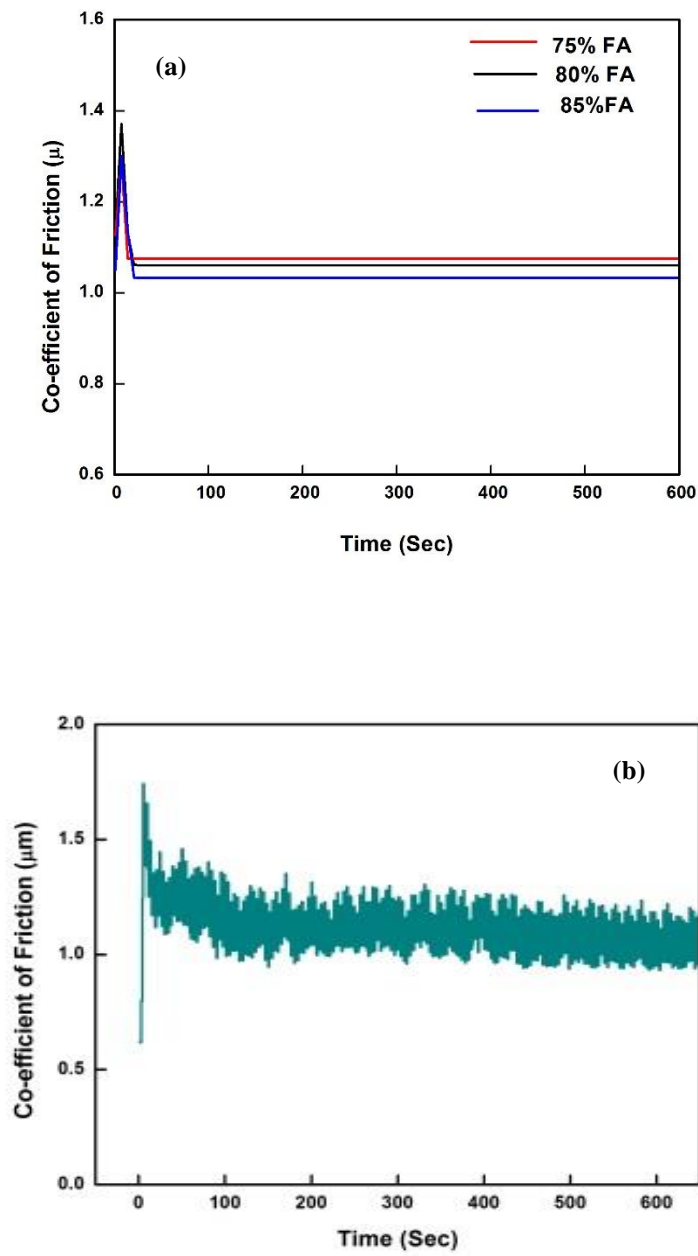


Fig.4.10 (a, b) Variation in co-efficient of friction w.r.t FA composition

## 4.10 Microstructural study of Fly ash polymer Composite

### 4.10.1 SEM Analysis

Microstructure of the composites with 75, 80 and 85 wt. % FA plus resin powder mix was studied by the SEM at different magnifications. Particle size of FA powder was also determined. It has been found that the particle size of FA lies in the range of 9.63- 47.6  $\mu\text{m}$ .

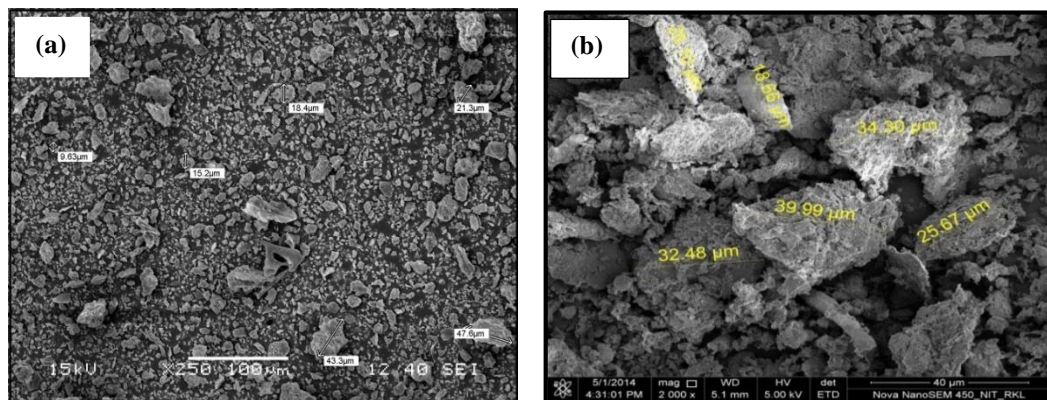


Figure.4.11 (a, b) Particle size distribution of FA powder at different Magnification

From the SEM micrographs it has been observed that FA particles are mostly spherical, messy and irregular in shape. FA particles are formed Coagulated junks. In case of 75 wt. % FA composition as shown in Figure 4.12 (a) the cracks at the inter particle boundary seems, pore type of interface periphery. With decrease in polymer addition i.e. increase in Fly ash amount it is seen that the interface bonding becomes better and less amount of cracks at the interfaces. With further decrease in resin addition, although there is good compaction but elongated cracks/cavities are found along the boundaries.

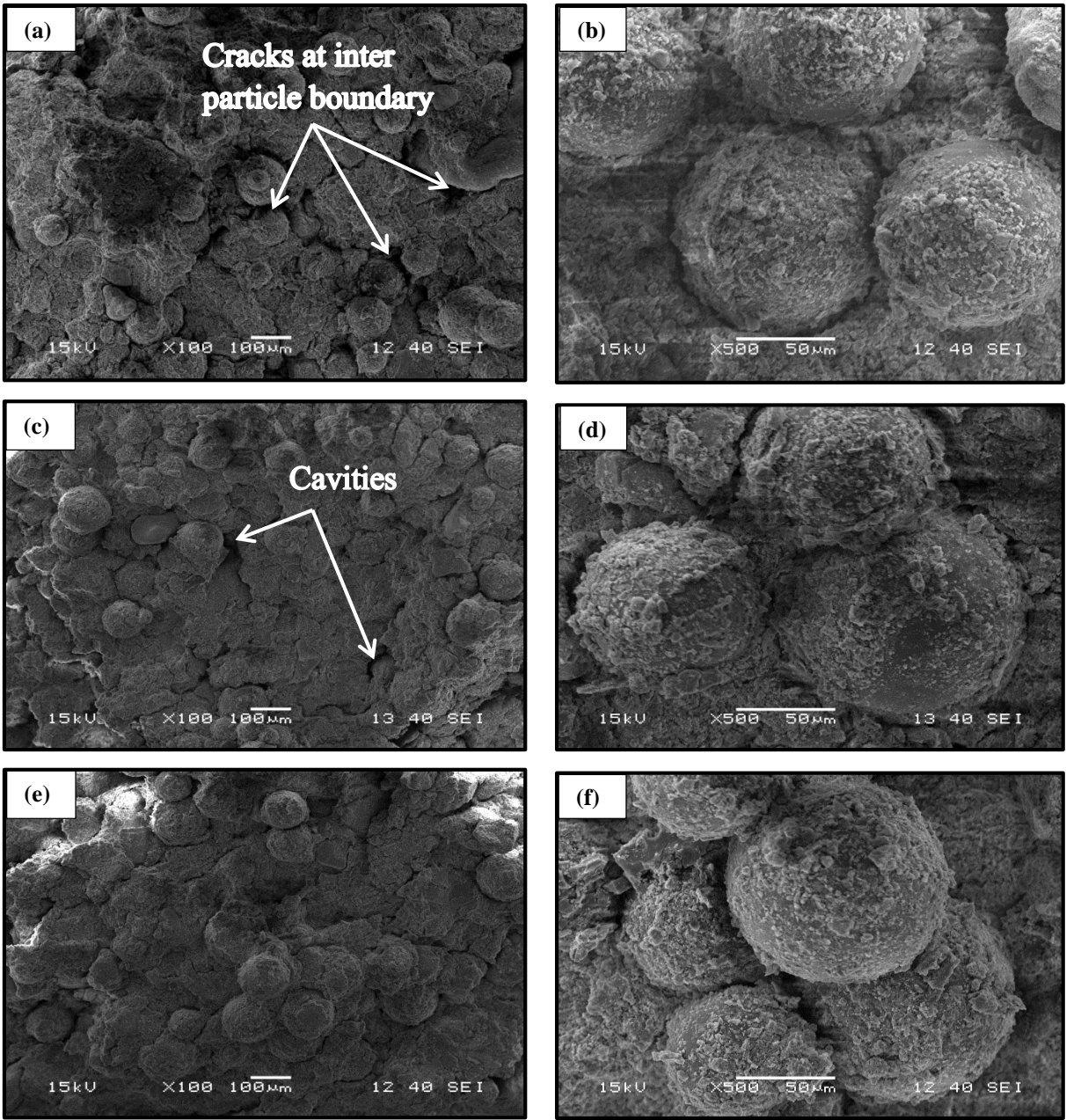


Fig.4.12 Morphology of Fly ash compacts with different composition and at different magnifications

Figure 4.13 shows the FESEM micrographs of wear track along sliding direction at different magnifications. These images show that wears mechanism is basically delamination, ploughing of the surface, formation of micro cracks and rubbing of tribolayer. Figure 4.13 (a) shows that Micro cracks have been initiated in direction perpendicular to sliding distance which leads to wear of the surfaces. Figure 4.13 (c) shows the wear track of dry compacts (80% FA composition) at very low magnification.

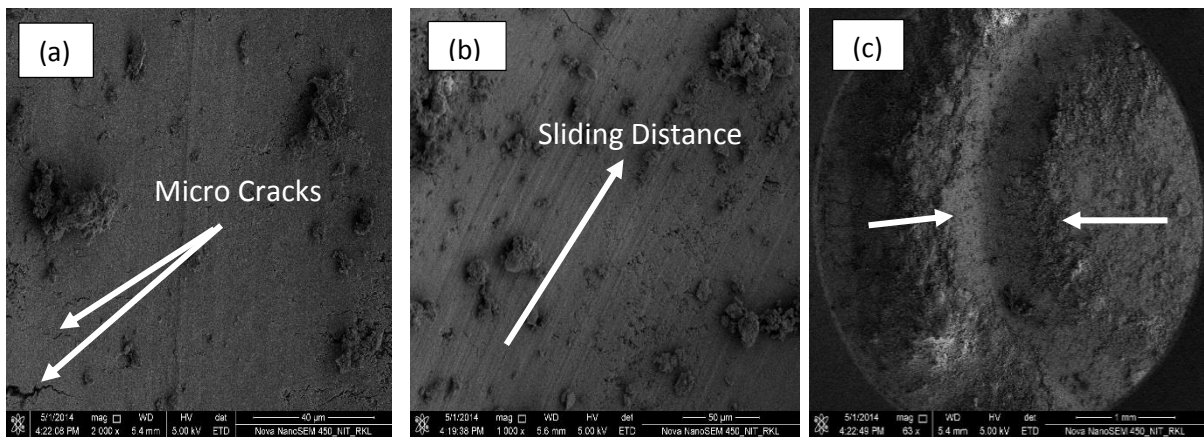


Fig. 4.13 FESEM image of wear track at different magnification

A blue geometric graphic consisting of several overlapping triangles and trapezoids, creating a dynamic, angular shape that points towards the right.

# CHAPTER 5

# Conclusions



## 5.1 Conclusions

On the basis of present study following conclusion can be drawn:

- 1) Water treated compacts shows positive effects on the hardness values. Out of all dry compacts, FA with 85 wt. % possesses a higher hardness value of 44.08 HV. Much improvement in the hardness value is achieved when the composites are treated in water at 110<sup>0</sup>- 180<sup>0</sup>C and this value rose to 47.37 HV. This increment in hardness value is due to the presence of CSH and CASH in the presence of moisture as obtained from XRD analysis.
- 2) With an increase in polymer addition (resin powder), the compressive strength of dry compacts decreases to a lower value of 6.5 MPa. Composition of 75 wt. % FA shows lower value. No significant reduction in Compressive strength is achieved in the case of wet compact.
- 3) Wear study of different composites can easily be correlated with the hardness value. In both the dry and wet state, FA with 85 wt. % composition shows better resistance to wear than other two compositions. Wear resistance increases with increase in FA content. The co-efficient of friction decreases with increase in FA percentage and follows a linear trend throughout the time of testing.
- 4) Thermal conductivity of FA increases with increase in temperature, whereas in case of resin powder FA mixes, the conductivity of composite decreases with increase in temperature. A much lower conductivity value is obtained and hence can be used as a substitute material with respect to clay.
- 5) Water absorption increases with increase in FA content. Maximum of 19% water is absorbed in case of 85 wt. % FA.
- 6) Density of dry compacts decreases with increase in FA content. While in case of wet compacts, it increases with increase in FA content.
- 7) SEM analysis revealed the morphology of FA particles that are mostly spherical in shape. With decrease in polymer addition i.e. increase in FA content the interface bonding becomes better and less amount of cracks were found at the interfaces.
- 8) XRD analysis revealed that FA particles mostly consist of Silica and alumina with less percentage of Fe<sub>2</sub>O<sub>3</sub>, Cao and others.

The Fly ash –resin powder composite produced in the present study seem to be appropriate for use as construction material. The production of this type of composite will certainly contribute to the use of fly ash for value added products. On the other hand, the reduction in clay usage for the production of conventional clay bricks will help to protect the environment.



## **CHAPTER 6**

# **References**

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